

## **TREATMENT OF MTBE USING FENTON'S REAGENT**

By

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### **ABSTRACT**

This paper addresses the removal of MTBE from water, using Fenton's Reagent. Although complete mineralization of MTBE by Fenton's Reagent was not achieved, greater than 99% destruction of MTBE was realized. This was accomplished at a  $\text{Fe}^{+2}:\text{H}_2\text{O}_2$  ratio of 1:1 and one hour of contact time. In all tests, twice the stoichiometric ratio of  $\text{H}_2\text{O}_2$  to MTBE was used. The major byproducts were tertiary butyl alcohol, tertiary butyl formate, and acetone with traces of 2-methyl-1-propene (isobutene). While small quantities of  $\text{O}_2$  evolved, no significant quantity of  $\text{CO}_2$  gas was detected.

### **INTRODUCTION**

Several oxygenates have been used in the United States since the 1970's as octane-enhancing replacements for lead tetraethyl. These include ethanol, methanol, ethyl tertiary butyl ether (ETBE), and tertiary butyl alcohol (TBA) as well as the currently controversial methyl tertiary-butyl ether (MTBE). Addition of MTBE to gasoline improves fuel combustion and reduces the resulting concentrations of carbon monoxide and unburnt hydrocarbons. The use of MTBE in gasoline at levels in excess of 10% by volume began in November 1992 when the requirements of the 1990 Clean Air Act Amendments (CAAA) mandated the use of oxygenated gasoline during the winter to help meet standards for carbon monoxide emissions. Furthermore, since January 1995, the CAAA also required nine metropolitan areas that have the most severe ozone pollution to use, year-round, reformulated gasoline that contains fuel oxygenates. Currently MTBE is added to about 30% of gasoline nationwide at an average concentration of about 11% by volume (USEPA, 1994). Since 1993, MTBE has been the second most produced organic chemical manufactured in the United States (USEPA, 1998). MTBE is the most commonly used fuel oxygenate because of its many favorable properties such as low production cost, ease of production, high octane rating, and favorable transfer and blending characteristics (Ainsworth, 1992; Shelly and Fouhy, 1994).

Unfortunately, the increased production and physical properties of MTBE have led to its introduction into the environment. It has been detected both in ground and storm waters. In a recent survey by the United States Geological Survey (USGS), MTBE was detected in 27

percent of the 210 wells and springs sampled, but none was found in drinking water wells (Squillace et al., 1996). Measurable concentrations of MTBE were also found in some of 592 storm water samples collected by the USGS in 16 cities and metropolitan areas required to obtain National Pollutant Discharge Elimination System (NPDES) permits (Delzer et al., 1996).

Physical and chemical properties of MTBE control its fate in the environment. MTBE, molecular weight 88, is a colorless liquid aliphatic ether with a characteristic odor. It gives water an unpleasant taste and odor at only a few tens of microgram per liter ( $\mu\text{g/L}$ ). It is highly soluble in water (about 48,000 mg/L) and has a low octanol-water partition coefficient ( $\log K_{ow} \sim 1.24$ ). Therefore, MTBE is highly mobile, undergoing little or no retardation as it travels through a groundwater system. Because of its very low Henry's Law Constant (0.022 at 25 degrees Celsius), almost 1/10th of benzene, it is difficult to remove MTBE from aqueous streams by air purging. It is resistant to biological degradation with a degradation half life of 10,000 days (27 years). Laboratory studies have shown that MTBE is resistant to all forms of aerobic as well as anaerobic biodegradation, key components in the natural attenuation process (Vance, 1998).

The U.S. Environmental Protection Agency (USEPA) has tentatively classified MTBE as a possible human carcinogen, but no drinking water regulation has yet been promulgated for MTBE (USEPA, 1997). However, a drinking water advisory of 20 to 40  $\mu\text{g/L}$  to avoid unpleasant taste and odor effects has been issued. This advisory concentration provides a large margin of safety for non-cancer effects and is in the range of margins typically maintained for potential carcinogenic effects (Squillace et al., 1998).

In July 1999, a USEPA advisory panel called for a substantial reduction in the use of MTBE as a gasoline additive and recommended that Congress remove the current requirement that 2% of reformulated gasoline by weight consist of oxygen - a mandate of the 1990 CAAA (Grisham, 1999).

MTBE's high solubility in water and recalcitrant characteristics make it difficult to remove from impacted water by conventional treatment technologies such as granular activated carbon (GAC), air stripping, or biological treatment. Previous research has demonstrated that Fenton's Reagent, a combination of hydrogen peroxide and ferrous sulfate, can effectively mineralize pure MTBE in water (Chen et al., 1995). Fenton's Reagent generates hydroxyl radicals, which are second only to fluorine in oxidation potential, and are capable of nonspecific oxidations (Bull and Zeff, 1992). The following equations represent the formation of hydroxyl radicals and complete oxidation of MTBE by Fenton's Reagent:



Under the sponsorship of USEPA's National Risk Management Research Laboratory (NRMRL), a bench-scale study was conducted by Battelle (Contract No. 68-C7-0008, Work Assignment

No. 1-11) to evaluate the use of Fenton's Reagent for treatment of MTBE in water.

## EXPERIMENTAL METHODS

### Materials

All chemicals used were obtained from Aldrich Chemical, Inc. and of ACS analytical grade or better. All solutions were prepared in deionized water (Millipore). All glasswares were rinsed, sequentially, with Alconox<sup>TM</sup> cleaning solution, 10% nitric acid, tap water, methanol, and deionized water (DI).

Stock solutions of acetone, MTBE, and tertiary butyl formate (TBF) each at 5,000 mg/L, and 1,000 mg/L of TBA were prepared in DI water. The stock solutions were stored in aluminum foil wrapped-volumetric flasks at 4°C. Calibration standards were prepared by serial dilution of the stock solutions.

Test samples for MTBE degradation studies were prepared from appropriate stock solution immediately before use.

A 5% H<sub>2</sub>O<sub>2</sub> solution was prepared by diluting 50% H<sub>2</sub>O<sub>2</sub> by DI water. Ferrous sulfate solution for Fenton's Reagent was prepared by dissolving FeSO<sub>4</sub>·7H<sub>2</sub>O solids in DI water.

Bovine catalase (Sigma) was used as received to help decompose excess H<sub>2</sub>O<sub>2</sub>.

### Analytical Procedure

Aqueous samples were analyzed (EPA 5021) with a Tekmar 7000 Headspace Analyzer<sup>TM</sup> equipped with a Varian Star 3400CX gas chromatography (GC) (Supelco SPB-1 60 meter long, 0.53 mm inside diameter, and 3.00 : m film fused silica capillary column) with a flame ionization detector (FID) and an auto-sampler. Samples were heated to 95°C for 55 minutes, agitated for 2 minutes, purged with helium into the GC column held at 35°C. With a 2-minute hold, the GC oven was programmed at 8°C/min to 150°C, then 10°C/min to 200°C.

A five-point calibration with concentrations of 0.5, 1, 5, 10, and 50 mg/L was performed for MTBE and TBA analyses. Triplicate samples were run for MTBE and TBA calibrations. A three-point calibration with 0.5, 1, and 5 mg/L was performed for acetone and TBF analyses. Duplicate samples were run for the acetone and TBF calibrations.

H<sub>2</sub>O<sub>2</sub> was analyzed using the Lamotte titration kit HP-5; residual H<sub>2</sub>O<sub>2</sub> after the reaction was determined using the Lamotte Octet Comparator test kit HP-40 after quenching with bovine catalase. Iron was determined using the Hach test kit following Standard Method 3500-Fe D.

For organic byproduct analysis, samples of the solution collected during the tests were analyzed by GC/mass spectrography (MS). The GC/MS consisted of a Hewlett-Packard (HP) GC with an HP 5970 mass selective detector and a Supelco SPB-1 column operating in full scan mode. In this mode, the mass spectrometer scanned all masses continuously between  $m/z$  30 and  $m/z$  300. Sample vials each containing 2-mL aqueous sample were heated in an oven to 60°C for 30 minutes. A sample was taken from the headspace of each vial and immediately injected into the GC/MS. Byproducts in the form of tentatively identified compounds (TICs) were confirmed using the National Bureau of Standards (NBS) data base. After by-products were identified by GC/MS, individual pure compounds were injected into the GC/FID and GC/MS for further confirmation of their retention times and mass spectra. After confirmation by both GC/MS and GC/FID, these compounds were calibrated on the GC/FID and quantified. Retention times of MTBE and associated byproducts are given in Table 1.

For gas analysis, the samples were analyzed using an SRI GC equipped with a CTRI concentric column connected to a thermal conductivity detector (TCD).

## Experimental Setup

Figure 1 shows the schematic diagram of the experimental apparatus used in this study. It consisted of a three-neck, 500-mL round bottom flask fitted with a pH meter, a sampling syringe, and a gas sampling assembly. The pH probe was inserted through one of the side necks into the test solution. The other side neck was fitted with a gas collection assembly consisting of a sampling port and a graduated burette designed to measure the volume of gas produced during the reaction. The center neck was fitted with a long needle syringe inserted through the septum. The needle was used to introduce nitrogen gas ( $N_2$ ) for flushing the reactor, to inject  $H_2O_2$  to begin the reaction, and to withdraw solution samples for chemical analyses. A magnetic stirrer was used to mix the test solution throughout the experiment.

## Experimental Procedure

Four experiments were conducted to accomplish the following three objectives:

1. identify and optimize operational parameters for treating MTBE contaminated water using Fenton's Reagent;
2. determine the percent destruction of MTBE; and
3. identify and quantify the possible reaction products.

Test conditions are tabulated in Table 2. For all tests,  $H_2O_2$  was added at two times the stoichiometric amount required for complete mineralization of MTBE (Eq. 2). The amount of ferrous sulfate added varied with each test and corresponded to the specific  $H_2O_2$ :Fe ratio.

For each test, 500 mL of MTBE test solution and a known amount of ferrous sulfate solid were quickly added to the flask through the center neck. After sealing the center neck with the septum, the solution was stirred until the ferrous sulfate solid was completely dissolved. The

tubing connected to the graduated burette was lifted up to bring the water level in the burette close to the top to minimize the head space in the burette. The valve at the bottom of the burette was then closed and the venting valve in the top of the burette was opened. The reactor was then flushed with ultra-pure  $N_2$  gas through the syringe for about 20 minutes. Subsequently, the burette valve was opened and the venting valve was closed. The water level in the burette was pushed down to the bottom of the burette before the valve on the  $N_2$  gas cylinder was turned off. A 10-mL gas mixture sample and a 10-mL aqueous sample were collected and then a known amount of  $H_2O_2$  was injected into the reactor to initiate the reaction. The reactor was sampled at time intervals following the initiation of the reaction.

Liquid samples were collected from the reactor vessel from the center neck using a glass syringe fitted with a 4-inch long needle. The sample was placed in 20-mL glass sample vial containing 5 drops of bovine catalase, which terminated the reaction by decomposing excess  $H_2O_2$ . Two 2-mL subsamples were transferred into two 20-mL auto-sampler vials, each containing 0.5 g NaCl, for duplicate head space analysis of MTBE by GC/FID. At the end of each test, the amount of  $H_2O_2$ , total Fe, and  $Fe^{2+}$  remaining in the solution were measured.  $H_2O_2$  was measured using a titration kit and iron was measured using Hack test kits.

Gas samples were collected from the gas sampling port using a 10-mL gas-tight syringe that was flushed twice with helium prior to use. Before the syringe was removed from the sampling port, the tubing connected to the burette was lifted to equalize water levels in the tubing and in the burette. The burette reading was recorded before and after each sampling event to calculate the volume of gas produced. Gas samples were analyzed for oxygen and carbon dioxide using a GC.

## RESULTS

The results of each test are summarized in Tables 3 through 6. During all tests, the solutions in the reactor (containing MTBE and  $Fe^{+2}$ ) turned yellow and turbid immediately after the addition of  $H_2O_2$ , indicating the formation of iron precipitates. The pH values of the solutions dropped rapidly after  $H_2O_2$  addition. Concentrations of MTBE decreased rapidly but stabilized within 5 minutes, indicating fast degradation kinetics (Tests #3 and #4). MTBE degradation products, 2-methyl-1-propene ( $C_4H_8$ ), acetone, TBA, and TBF were identified in all samples collected during the tests. But the amounts of gases produced during these tests were not quantified. Although some  $O_2$  was evolved, no detectable quantity of  $CO_2$  was found.

Effects of  $H_2O_2:Fe^{+2}$  ratios on MTBE reduction, after 60 minutes of reaction, are shown in Table 7. Percentage reduction of MTBE of > 99, 97, 66, and 0 were achieved at  $H_2O_2:Fe^{+2}$  ratios of 1:1, 6:1, 10:1, and 100:1, respectively. As the  $Fe^{+2}$  dose increased (i.e., the  $H_2O_2:Fe^{+2}$  ratio was decreased), the percentage of MTBE degradation also increased.

## CONCLUSIONS

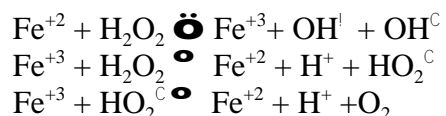
Oxidation of MTBE showed that the main reaction products were: acetone, TBA, and

TBF. Small quantities of 2-methyl-1-propene (isobutylene) (C<sub>4</sub>H<sub>8</sub>), a gas of molecular weight 56 and insoluble in water, were found, but due to their small quantities were not quantified. Also, no CO<sub>2</sub> was observed. Quantities of oxygen evolved were also minimal. These results indicate that the degradation of MTBE by the classical Fenton's Reagent as represented by the equation: C<sub>5</sub>H<sub>12</sub>O + 15 H<sub>2</sub>O<sub>2</sub> → 5CO<sub>2</sub> + 21 H<sub>2</sub>O was not realized under the experimental conditions of this study.

In these oxidation reactions, the amounts of MTBE used were at or below 21 mg/L. Even at these low concentrations, under optimum reaction conditions, the residual concentrations of MTBE, after 1 hour of contact, were below 2 mg/L. These results show that Fenton's Reagent can reduce the concentrations of aqueous MTBE well below the regulated limit. Other important findings of the study are:

1. The optimum ratio (molar) of H<sub>2</sub>O<sub>2</sub> to Fe appears to be 1:1. At this ratio more than 99% removal of MTBE was achieved at a contact time of only one hour. This ratio of hydrogen peroxide to iron is the recommended ratio for preparing Fenton's Reagent and it is no surprise that the highest reduction of MTBE was achieved at this ratio.

The classical Fenton's reaction consists of following steps:



These equations explain why a large excess of either iron or hydrogen peroxide does not help in the destruction of MTBE.

2. After one hour of contact, the initial pH of the mixtures dropped from about 5 to 3 signifying the formation of organic acids. The formation of TBF supports this contention.

A cursory look at the results of the two tests (#1 and #4) which gave high percentage of MTBE removal, showed that input of MTBE (as carbon) and the products (calculated as carbon) do not match. As a matter of fact, close to 80 and 60 percent of the product were missing for Tests 1 and 4, respectively (Table 8). This may be due to the formation of some volatile products which are escaping the reaction system or the generation of some nonvolatile organics, for example, formic and/or acetic acids.

In an ongoing study (Contract No. 68-C98-157, Work Assignment No. 0-2), an attempt will be made to resolve these unanswered questions.

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**Table 1. GC Retention Time of MTBE and Associated Degradation Byproducts**

Compounds	Retention Time (minutes)
2- methyl-1-propene	2.5
Acetone	3.3
TBA	3.9
MTBE	4.9
TBF	6.4

**Table 2. Test Conditions for MTBE Degradation**

Chemical Addition	Unit	Test 1	Test 2	Test 3	Test 4
20 mg/L MTBE solution	mL	300	300	500	500
5% H <sub>2</sub> O <sub>2</sub> solution	mL	1.2	1.2	2	2
Ferrous sulfate solid	mg	568	5.68	96	158
H <sub>2</sub> O <sub>2</sub> :Fe <sup>+2</sup>	mole:mole	1:1	100:1	10:1	6:1
Test duration	hr	1	4	4	4

**Table 3. MTBE Degradation Test 1 Results ( $\text{H}_2\text{O}_2:\text{Fe}^{+2} = 1:1$ )**

Time (min)	Aqueous Sample Measurement								Gas Sample Measurement		
	pH	MTBE (mg/L)	Acetone (mg/L)	TBA (mg/L)	TBF (mg/L)	$\text{H}_2\text{O}_2$ (mg/L)	Total Fe (mg/L)	Soluble Fe(II) (mg/L)	$\text{O}_2$ (%)	$\text{CO}_2$ (%)	Gas Production (mL)
0	4.6	21.0 21.2	ND 0.1	0.1 0.1	0.2 ND	236	NA	NA	0.28	ND	NA
30	2.49	0.15 0.1	3.9 3.7	1.0 0.9	0.8 0.7	156	NA	NA	1.89	ND	ND
60	2.47	0.1	3.8	0.7	0.1	125	NA	NA	1.89	ND	ND

**Table 4. MTBE Degradation Test 2 Results ( $\text{H}_2\text{O}_2:\text{Fe}^{+2} = 100:1$ )**

Time (min)	Aqueous Sample Measurement								Gas Sample Measurement		
	pH	MTBE (mg/L)	Acetone (mg/L)	TBA (mg/L)	TBF (mg/L)	$\text{H}_2\text{O}_2$ (mg/L)	Total Fe (mg/L)	Soluble Fe(II) (mg/L)	$\text{O}_2$ (%)	$\text{CO}_2$ (%)	Gas Production (mL)
0	4.96	19.6 16.9	ND ND	ND ND	ND ND	236	NA	NA	ND	ND	NA
30	3.96	22.6 18.2	0.2 0.2	0.5 0.4	1.6 1.3	NA	NA	NA	0.4	ND	ND
60	3.98	19.5 21.1	0.2 0.2	0.5 0.5	1.3 1.5	NA	NA	NA	NA	ND	ND
150	3.98	18.0 20.6	0.2 0.2	0.6 0.7	1.2 1.5	NA	NA	NA	0.36	ND	ND
240	4.08	19.1 18.2	0.2 0.2	0.8 0.7	1.4 1.3	>200	3.2	ND	0.52	ND	ND

**Table 5. MTBE Degradation Test 3 Results ( $\text{H}_2\text{O}_2:\text{Fe}^{+2} = 10:1$ )**

Time (min)	Aqueous Sample Measurement								Gas Sample Measurement		
	pH	MTBE (mg/L)	Acetone (mg/L)	TBA (mg/L)	TBF (mg/L)	$\text{H}_2\text{O}_2$ (mg/L)	Total Fe (mg/L)	Soluble Fe(II) (mg/L)	$\text{O}_2$ (%)	$\text{CO}_2$ (%)	Gas Production (mL)
0	5.41	18.3 16.9	ND ND	ND ND	ND ND	236	NA	NA	0.87	ND	NA
5	3.17	6.6 6.2	2.3 2.0	4.9 4.4	6.9 5.6	NA	NA	NA	NA	NA	ND
10	3.13	6.4 6.2	2.3 2.1	4.8 4.3	6.9 6.9	NA	NA	NA	NA	NA	ND
30	3.1	6.6 6.4	2.2 2.2	4.6 4.6	6.9 6.4	NA	NA	NA	NA	NA	ND
60	3.07	6.2 5.9	2.2 2.0	4.6 4.3	6.0 5.6	NA	NA	NA	NA	NA	ND
120	3.04	5.8 5.7	2.4 2.3	4.6 4.5	5.2 5.4	NA	NA	NA	NA	NA	ND
240	3.01	5.1 5.1	2.4 2.3	4.5 4.4	5.5 5.6	200	3.2	ND	0.67	ND	ND

**Table 6. MTBE Degradation Test 4 Results ( $\text{H}_2\text{O}_2:\text{Fe}^{+2} = 6:1$ )**

Time (min)	Aqueous Sample Measurement								Gas Sample Measurement		
	pH	MTBE (mg/L)	Acetone (mg/L)	TBA (mg/L)	TBF (mg/L)	$\text{H}_2\text{O}_2$ (mg/L)	Total Fe (mg/L)	Soluble Fe(II) (mg/L)	$\text{O}_2$ (%)	$\text{CO}_2$ (%)	Gas Production (mL)
0	5.08	17.4	ND	0.1	ND	236	NA	NA	ND	ND	NA
5	2.99	0.7 0.3	1.8 0.9	1.5 0.7	3.7 1.7	NA	NA	NA	NA	NA	ND
10	2.96	0.6 0.4	1.7 1.1	1.4 1.1	2.9 2.2	NA	NA	NA	NA	NA	ND
30	2.93	0.5 0.6	1.7 1.8	1.4 1.7	3.0 3.1	NA	NA	NA	NA	NA	ND
60	2.91	0.5 0.5	1.8 2.0	1.3 1.6	2.9 2.7	NA	NA	NA	NA	NA	ND
120	2.88	0.3 0.3	1.9 2.1	1.1 1.3	2.1 2.3	NA	NA	NA	NA	NA	ND
240	2.86	0.2 0.2	2.1 2.2	1.0 1.1	1.8 1.8	185	6.8	0.2	1.3	ND	ND

**Table 7. Reactions of Aqueous MTBE with Fenton's Agent Under Several Conditions**  
**(All data are after 60 minutes of reaction. All concentrations are in mg/L)**

Subject	Test #1	Test #2	Test #3	Test #4
Molar ratio MTBE: Fe	1:29	1:34.7	1:34.7	1:34.7
Molar ratio H <sub>2</sub> O <sub>2</sub> : Fe	1:1	100:1	10:1	6:1
Input MTBE (mg/L)	21.10	18.25	17.60	17.40
Residual MTBE (mg/L)	0.10	20.30	6.05	0.50
Initial pH	4.60	4.96	5.41	5.08
Final pH	2.47	3.98	3.07	2.91
% MTBE removal	>99	0	66	97
%H <sub>2</sub> O <sub>2</sub> * utilized	47	< 15	15	22
Acetone (mg/L)	3.80	0.20	2.10	0.50
TBA (mg/L)	0.70	0.50	4.45	1.45
TBF (mg/L)	0.10	1.40	5.80	2.80

\* calculated on the basis of input and outputs of hydrogen peroxide

**Table 8. Mass Balance of Test #1 (~100% MTBE Removal) and Test #4 (>97% MTBE Removal)**

Input	Test #1 21.1 mg/L MTBE=14.36mg/L 'C'	Test #4 17.4 mg/L MTBE = 11.83 mg/L 'C'
Output	0.1 mg/L MTBE=0.07 mg/L 'C' 3.8mg/L acetone=2.36 mg/L 'C' 0.7 mg/L TBA = 0.46 mg/L 'C' 0.1mg/L TBF = 0.06 mg/L 'C' Total = 2.95 mg/L 'C'	0.5 mg/L MTBE = 0.34mg/L 'C' 1.90 mg/L acetone = 1.18 mg/L 'C' 1.45 mg/L TBA = 0.94 mg/L 'C' 2.80 mg/L TBF = 1.62 mg/L 'C' Total = 4.08 mg/L 'C'
% missing	79.5	65.5

Note: 'C' = as carbon.

In either of these tests, no CO<sub>2</sub> or any volatile organic compound was detected.

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